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Preparation and properties of mechanically alloyed and electrochemically etched porous Ti–6Al–4V

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ABSTRACT

Formation of porous Ti–6Al–4V nanostructure biomaterial was described. The alloy was prepared by mechanical alloying followed by pressing, sintering and subsequent anodic electrochemical etching in 1 M H_3PO_4 + 2% HF electrolyte at 10 V for 30 min. Mechanically alloyed Ti–6Al–4V has nanostructure with grain size of about 35 nm and large grain boundaries volume fraction, which essentially improve etching process. The electrolyte penetrates sintered compacts through the grain boundaries, resulting in effective material removing and pores formation. The pore diameter reaches up to 60 μm, which is very attractive for strong bonding with bone. The anodization of the microcrystalline alloy ingot results in selective etching, revealing of the two-phase structure with relatively flat surface. The corrosion properties were investigated in Ringer's solution. Mechanically alloyed samples shows worse corrosion resistance than the bulk microcrystalline alloy ingot, but electrochemical etching results in improving corrosion resistance.

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1. Introduction

The Ti–6Al–4V was one from the firstly used Ti-based biomedical alloys. This alloy shows good mechanical and chemical properties, respective for implant applications [1–4].

Ti-based alloys generally have excellent properties, but for the hard tissue replacement application, an additional surface treatment improving surface roughness is required. The rough surface is attractive for the osseointegration process, which results in fast tissue growth and strong bone bonding with implant. The implant surface can be modified using mechanical, chemical, electrochemical or other methods resulting in pits and pores formation [5–8]. The simple electrochemical etching is a useful method for the roughness as well as corrosion resistance improvement, due to anodic oxidation conditions [9].

The anodic etching results in surface roughening related with pits or pores formation. The micrometers pores results in easy tissue growth and strong fixing of the implant with bone. The etching usually proceeds in phosphoric acid solutions [7]. Alternatively for the electrochemical etching, the powder metallurgical process can be successfully applied for the microporous Ti-based implant preparation [10].

Formation of nanostructure in Ti-based alloys for implant applications should be useful for increase of mechanical properties and large grain boundaries volume fraction states an easy way for the diffusion and penetration by electrolyte, resulting in effective etching [11]. In this work was studied the formation of porous Ti–6Al–4V structure using both mechanical alloying and electrochemical etching.

2. Experimental

Bulk Ti–6Al–4V microcrystalline alloy was used for the etching as a reference material. As a new approach, mechanical alloying was applied for the preparation of the nanocrystalline alloy, followed by pressing, sintering and electrochemical etching, respective for the surface roughening.

In the mechanical alloying, pure elements Ti, Al, V (Sigma–Aldrich), with grain size of about 300 mesh were mechanically milled in protective high purity argon atmosphere for 48 h, using SPEX 8000 mill. After the process, the powder handled in MBraun Labmaster glove box with argon atmosphere, was uniaxially pressed at 400 MPa to obtained compacts with diameter and height of 8 and 5 mm, respectively. After the compacting, the green compacts were sintered at 1000 °C for 60 min in 95% Ar + 5% H_2 atmosphere. In the following stage the electrochemical etching was done using 1 M H_3PO_4 + 2% HF electrolyte at 10 V vs. OCP for 30 min, using Solartron 1285 potentiostat equipped with electrochemical cell. Structure and grain size was identified by XRD diffractometer. Morphology and composition was identified by Vega Tescan SEM, equipped with PGT Prism EDS.

Corrosion resistance was measured using potentiodynamic mode in EG&G corrosion cell, with scan rate fixed to 0.5 mV/s.

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Ringer's solution (NaCl – 9 g/l, KCl – 0.42 g/l, $CaCl_2$ – 0.48 g/l, $NaHCO_3$ – 0.2 g/l) was used as the simulated body fluids.

3. Results and discussion

Mechanical alloying is a simple and cheap process for nanomaterials synthesis. In the process, the mixture of microcrystalline pure elemental powders (Fig. 1a) in the stoichiometric ratio was milled for 48 h to achieve Ti–6Al–4V alloy composition. The XRD data in Fig. 1b clearly shows that after milling, the Ti–Al–V mixture is ultra fine grained (18 nm). After sintering (Fig. 1c) the structure is comparable to the microcrystalline bulk alloy (Fig. 1d), but with definitely smaller grains (35 nm). Due to structure, this material exhibit quite different properties. Generally mechanical alloying consists of repeated fracture, mixing and cold welding of a fine blend of elemental particles, resulting in size reduction and chemical reactions.

The sintering results in compacts, used for the next treatment, respective for the pores formation. The compacts with relatively low density (90% of the theoretical density) are attractive material for the electrochemical etching, due to the large volume of the grain boundaries, which state the easy paths for the electrolyte penetration. For that reason the nanocrystalline compacts were etched fast and easy. The applied electrolyte contained H_3PO_4 with small HF addition, which enhanced dissolution [9] and results in porous Ti–6Al–4V compacts (Fig. 2a and b). After pressing, sintering and electrochemical etching, the nanocrystalline compacts achieved density of about 80% of the bulk ingots, which is related to pores in the compacts. The electrolyte composition as well as etching conditions works also very well during the etching of pure microcrystalline Ti as well as Ti/ceramic nanocomposites [9,11], but for microcrystalline Ti–6Al–4V alloy ingots does not work

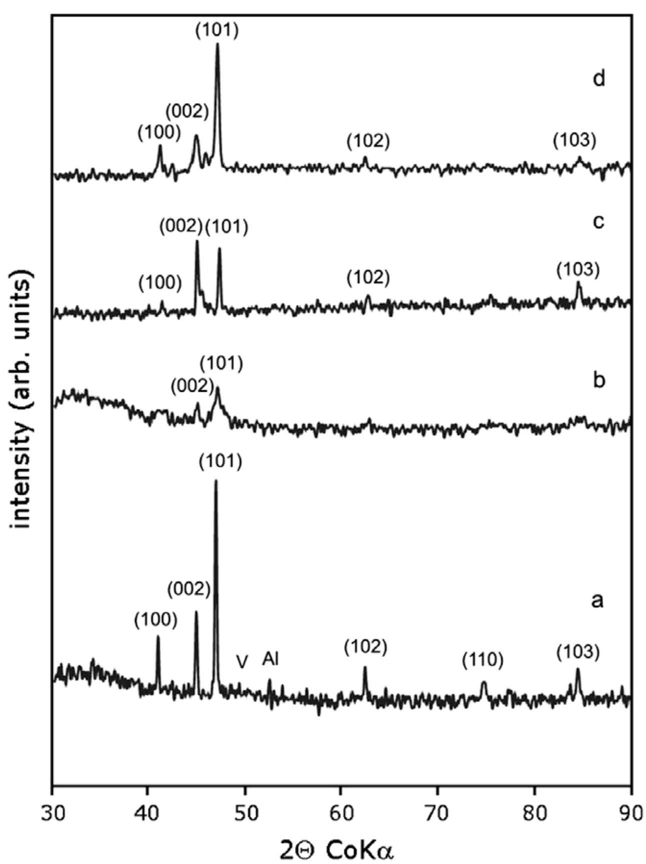

Fig. 1. X-ray data for mixture of the microcrystalline Ti, Al and V powder before MA (a) after MA (b), after sintering (c) and for comparison bulk microcrystalline Ti–6Al–4V alloy (d).

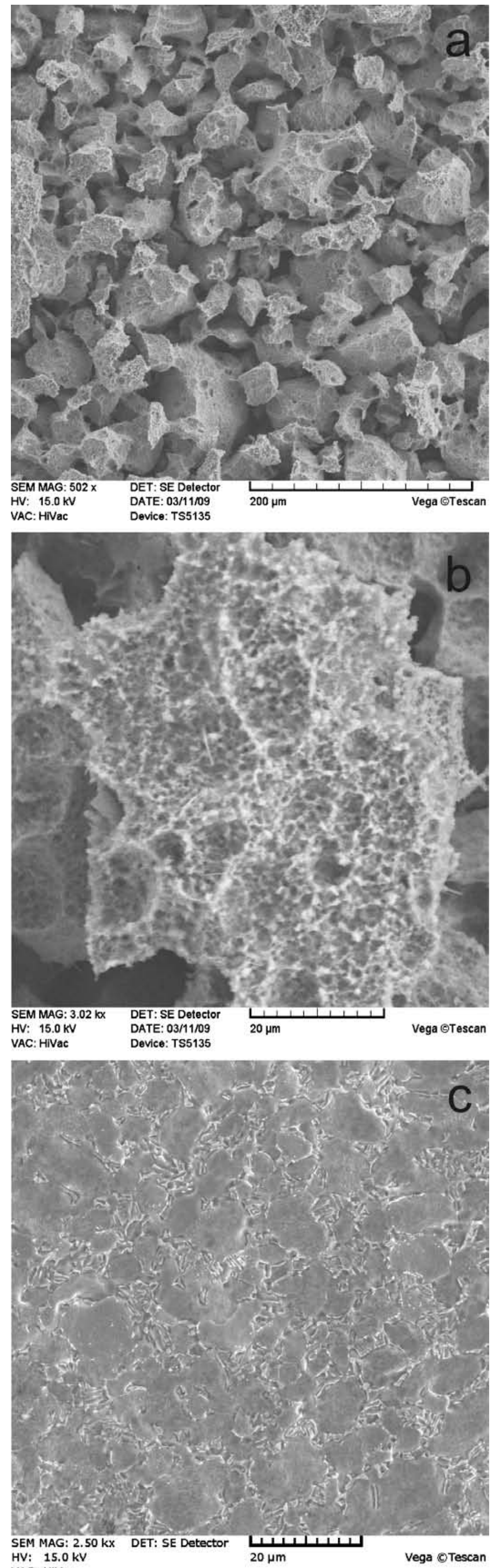

Fig. 2. Mechanically alloyed Ti–6Al–4V after sintering and electrochemical etching (a, b); for comparison is shown electrochemically etched bulk microcrystalline sample (c).

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